PATENT ABSTRACTS OF JAPAN

(11)Publication number :

09-012860

(43)Date of publication of application: 14.01.1997

(51)Int.Cl.

CO8L 71/02 CO8K 5/09 C08K 5/17 C08L101/10

(21)Application number: 07-164188

(71)Applicant : ASAHI GLASS CO LTD

(22)Date of filing: 29.06.1995

(72)Inventor: WATABE TAKASHI

YAMAMOTO HIROTSUGU

DOI TAKAO

(54) ROOM-TEMPERATURE-CURABLE COMPOSITION

(57)Abstract:

PURPOSE: To obtain a room-temp.-curable compsn. which exhibits a sufficient depth cure rate and is free from troubles. such as the viscosity increase of the main component, even during long stroage by compounding a specific org. polymer with a cure catalyst, an org. amine compd., and a filler. CONSTITUTION: This compsn. is prepd. by mixing and kneading 100 pts.wt. org. polymer which is derived from a hydroxylated polyoxyalkylene polymer obtd. by polymerizing an alkylene oxide using an initiator in the presence of a composite metal cyanide complex as the catalyst and has hydrolyzable silicon groups represented the formula (R1 is a 1-20C monovalent hydrocarbon group, R2 is a divalent org. group; X is a hydroxyl or hydrolyzable group; and a is 1-3) and a total content of ionic impurities of 50ppm or lower with 0.01-10 pts.wt. cure catalyst selected from among 20C or lower aliph. carboxylates of divalent tin, divalent bismuth, and divalent lead, 0.01-10 pts.wt. org. amine compd., 50-250wt.% (based on the polymer) filler, and optional plasticizers and additives.

 $-R^2 - SiX_x R^{1}_{3-x}$

* NOTICES *

JPO and IMPIT are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.

2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

CLAIMS

(in Ultimical)

[Claim, (a) Line gatalyst. (B) chosen from the following organic polymer (A), carboxylate of disaltert
the carboxylate of disaltert birmuth, and carboxylate of disaltert lead, and an organic annie ocompound
that a noral-measure—curing mitter one contentent containing to builty agent (D) (Cognosio
polymer) it is derived from a hydroxyl group content polyonylakylate polymer (E) produced by

polymerizing alkylene oxide in an initiator by making a oomposite metal cyanide complex (E) into a catalyst, An organio polymer (A) whose total amount of an ionio impurky it has a hydrotytic silicon

-R²-SiX_aR¹ 3-a ... (1)

group expressed with a formula (1), and is 50 ppm or less.

ha for \mathbb{R}^1 , a hydroxyl group or a hydrolytic basis, and a of substitution of the carbon numbers 1–20 or an unsubstituted univalent hydrocarbon group, and \mathbb{R}^2 are the integers of 1–3 a divalent organic group

Claid X among a formula.

The formula and a formula and a

(Jolian 3(John 1 or 2 room-tempersture-curing nature constituents which are a compound in which an organic amine compound (c) has a hydrolytic silicon group expressed with an amino group and a formula (2) to internolecular.

-R3-SiX1 bR13-b ... (2)

As for a divalent organic group and R², a hydroxyl group or a hydrolytic basis, and b of substitution of the carbon numbers 1-20 or an unsubstituted univalent hydrocarbon group, and X¹ are [R²] tha thegars of 12 among a formula musual parties or another of either of the Olaims 1-3 which is an ionic (Jaim 4.4) room-removerfular-curing nature constituent of either of the Olaims 1-3 which is an ionic

impurity in which an ionio impurity contains metallic compounds and/or an alkali metal compound

exabiliting from a composite metal systation complexe (E). (Claim Spike or accomposite metal systation contained in a polymer (F). After an ionic impossity contained in a polymer (F) after considering it as a polymer to station or spoke (F) after considering it as a polymer (F) after considering it as a polymer to station or (F) by removing this safe from a polymer (F) after considering it as a polymer to as an incubble as the fritting in the station of the station o

(Claim (BA) or openior in one a polymer (A) an onic imparity contained in a terminal unsaturated group (Claim (BA) or openior (A) or a horizon or a polymer (A) and one of the contract of the contract or an openior or a contract or an openior openior openior or an openior ope

HSIX_R¹ ²⁻² ... (3)
A hydroxy large or a hydrolytic basis, and a of R¹ are the integers of 1–3 among a formula
A hydroxy large or or a hydrolytic basis, and a of R¹ are the integers of 1–3 among a formula
A hydroxy large or a hydrolytic basis, and a of R¹ are the integers of 1–3 among a formula

Claim 7]A room-temperature-curing nature constituent of either of the Claims 1-6 whose hydroxyl

http://www4.ipdl.inpit.go.jp/ogi-bin/tran_web_ogi_ejje?atw_u=http://www4.ipdl.inpit.go.jp... 2010/04/30

JP,09-012860,A [CLAIMS]

value conversion molecular weights of a polymer (F) are 5000-30000.

[Translation done.]

http://www4.ipdl.inpit.go.jp/cgi-bin/tran_web_cgi_ejje?atw_u=http://www4.ipdl.inpit.go.jp... 2010/04/30

NOTICES *

smages expeed by the use of this translation. PO and INPIT are not responsible for any

This document has been translated by computer. So the translation may not reflect the original

**** shows the word which can not be translated. In the drawings, any words are not translated.

DETAILED DESCRIPTION

Detailed Description of the Invention

Industrial Application]This invention relates to the room-temperature-ouring nature constituent nardened under hygroscopic-surface-moisture existence. 0002

and which is known as conventional, for example, modified silicona, system rasin is known well, and is Description of the Prior ArdTha method of using it for a sealing material, adhosives, eto. using the nardaning reaction of various kinds of organic polymera which have a hydrolytic allioon group at the

Problem(s) to be Solved by the Invention|The organic polymer which has a hydrolytic silicon group at such the end is proposed by JP,45–36319,B, JP,46–17553,B, JP,61–18582,B, etc., for example.

usaful method industrially.

0004]in order to give room-temperature-ouring nature to an end especially with the organic polymer which has alkoxy sityl groups as a hydrolytic silicon group among the organic polymers which have a ydrolytic silicon group, using what is called a curing catalyst is usually performed. As such a curing

(0005)To the organic polymer which has a hydrolytic silicon group at the end, various bulking agents, catalyst, organic metallic compounds such as metal salt of carboxylic acid, acidity, or a basic compound is known, and carboxylate of tin and other organotin compounds are especially common. a plasticizer, it is divided roughly into 2 gestalten called what is called a 1 liquid type and a 2 liquid ype as a gestalt of the combination when using the resin composition of the room-temperaturecuring nature which blends an adhesive grant agent, a thixotropy grant agent, a curing catalyst, various stabilizer, etc. for a sealing material, adhesives, etc.

t mixes colorant etc. with these two ingredients if needed furthar, constructs, and makes it harden in divalent tin and the organic amine compound, and the method of specifically using octylic acid tin and 0006]What is known as a 2 liquid type among those is divided into base resin and a hardening agent. a service space at the time of use. As for the hardening agent in this 2 liquid type, it is common to compression restoration nature to a repetition of elasticity, leaden oarboxylate or carboxylate of nolude the ouring catalyst, and as this curing catalyst, Since a hardened material shows good auryl amine are proposed (refer to JP.61-60867,B).

Were manufactured by the method of introducing a hydrolytic silicon group, after a dihalo compound's which have a hydrolytic silloon group are used as base resin, by a part far from the surface of a cure [0007]However, in a 2 liquid type, as an organic polymer which has an end hydrolytic silicon group, . rate, especially a oured body, the so-called oure rate of the depths is insufficient, and it cannot be aforementioned well-known example, setting it and carrying out Polymer Division quantification. If ootylio acid tin and lauryl amine are used as a curing catalyst when the polymer and bulking agent thing the polyether compound of the comparatively short molecular weight proposed by the

JP.9-79627.A is used as base rusin, in order not to usually parform striot moisture control (that is, let te amount of [in base resin] watar contant fall) when manufacturing base resin although the (0008].When the organic polymer containing the hydrolytic silicon group manufactured by the end complax given [as an organic polymer which has a hydrolytic silicon group] in JP,3-43449,A and rom the polyoxyalkylene polymer which manufactured as a catalyst the composita metal cyanide ardenability of the depths improves remarkably if this curing catalyst is used, there was a casa ttp://www4.ipdl.inpit.go.jp/cgi-bin/tran_web_cgi.ejje?atw_u=http%3A%2F%2Fwww4.ipdl.i. 2010/04/30

JP,09-012860,A [DETAILED DESCRIPTION]

where the viscosity of base resin rose during long-term preservation, and use top inconvenience

Meana for Solving the Problem]This invention is the following invention which is going to cancel such 0010] A curing catalyst (B) chosen from the following organic polymer (A), carboxylate of divalant tin,

carboxylate of divalent bismuth, and carboxylate of divalent lead, and an organic amine compound (C) produced by polymerizing alkylene oxide in an initiator by making a composite metal cyanide complex (E) into a catalyst, An organic polymer (A) whose total amount of an ionic impurity it has a hydrolytic silicon group expressed with a formula (1), and is 50 ppm or less. 0011](Organic polymer) It is derived from a hydroxyl group content polyoxyalkylene polymer (F) and a room-temperature-curing nature constituent containing a bulking agent (D).

0012]-R2-SIX₈R1 3-3 ... (1)

[0013]As for R1, a hydroxyl group or a hydrolytic basis, and a of substitution of the carbon numbers 1–20 or an unsubstituted univalent hydrocarbon group, and \mathbb{R}^2 are the integers of 1–3 a divalent

0015]By using a composite metal oyanide complex (E), using the conventional alkaline metal cetalyst, polyoxyalkylene polymer (F) produced by polymerizing alkylane oxide in an initiator by making a 0014][Organio polymer] An organio polymer (A) is derived from a hydroxyl group content composite metal oyanide complex (E) into a catalyst. organic group and X among a formula.

hypoxiacosity can be obtained more in the amount of Polymer Division.

([0] (0] A complex (2) is preferred, and the either supercologation as the arm ingredients as a composite metal oyanise complex (3) is preferred, and the either sand/or especially an alcoholio complex are preferred, oyanise complex (3) is preferred, and the either sand/or especially an alcoholio complex are preferred.

M.../M., is [rather than] small and a hydroxyl group content polyoxyalkylene polymer (F) of

The presentation can use what is intrinsically indicated to JP,S46-27250,B. As ether, ethyleneglycol dimethyl ether (gyme), diethylans giyool dimethyl ether (jig lime), eto, are preferred, and especially glyme is preferred from handling at the time of manufacture of a complex. As alcohol, t-butanol is

preferred 2-8 pieces. Specifically Ethylene glycol, a diethylene glycol, propylene glycol, Dipropylene pentaerythritol, digiycerol, a shook sirloin, and these. One-sort single use or two or more sorts of compound is preferred, and a polyhydroxy compound which has 2-4 hydroxyl groups especially is głycol, neopentyl glycol, 1,4-butanediol, There is polyol of low molecular weight from an object 0017]A compound which has 2-10 active hydrogen as an initiator is preferred, a polyhydroxy produced by making alkylene oxide react to 1,6-hexanediol. glycerin, trimethylolpropane,

0018]A total amount of an ionio impurity of an organic polymar (A) of this invention is 50 ppm or metallic compounds and/or an alkali metal compound resulting from a composite metal cyanide ass. This invention is suitable when aspacially an ionic impurity is an ionic impurity containing concomitant use may be sufficient as these. An unsaturation group content mono-hydroxy compound like allyl alcohol can also be used.

.0019]By reducing such metal impurity quantity, the storage stability of an organic polymer (A) and a when removing metallic compounds resulting from especially a composite metal cyanide complex (E) [0020]Following (I) - a method of (III) are mentioned as the reducing method. Methods of using it, nardenability constituent of this invention is improved more, and also hardenability which was oomplex (E). It is preferred that ionic impurities are 30 ppm or less and 20 more ppm or less. excellent in order not to bar an operation of a curing catalyst is acquired.

an insoluble saft to a polymer intrinsically, by drying, a salt is deposited and there is a method of next include (III). (I) Especially since a method can reduce an ionic impurity effectively and economically, it phosphoric acid, pyrophoric gold, disodium dihydrogen pyrophosphate, atc. are preferred. A deposited agent [a compound, water, and if needed] which specifically react to an ionic impurity and can form (0021)(0) How to remove this salt from a polymer after making into an insoluble salt intrinsically an ionic impurity contained in a polymer to a polymer. After adding the Nonion system surface-active removing this salt. As a compound which can form a salt, chloride, sulfuric soid, nitric soid,

http://www4.ipdl.inpit.go.jp/cgi-bin/tran.web_ogi.ejie?atw_u=http%3A%2F%2Fwww4.ipdli.. 2010/04/30

salt is removable by filtration operation, adsorbing operation, atc.

0023]To a buffer for pH and arbitration, by ammonia and a chelating agent (III) After processing, A fter adding a mathod and fatty alcohol from which metallic compounds resulting from a composite nethod of removing metallic compounds which originate in a composite metal cyanide complex (E) compounds which originate in a composite metal evanide complex (E) after processing with an netal oyanide complex (E) are removed, and a cheleting agent, a method of removing metallic in jonio impurity after adding a solvent to a polymer.

syanide complex (E). A cation and anions, such as zinc ion, cobalt ion, cyanide ion, and a chloride ion. Mix as an impurity at a process of manufacturing an organic polymer (A). Sodium ion, alkali metal ion process of manufacturing a halogen ion; organicity polymer (A); An organic polymer (A) at a process [0024]. With an ionic impurity as used in the field of this invention, originate in a composite metal like potassium ion, carboxylate ion that polyoxyalkylene generated in response to oxidation at a to manufacture An ester bond, All the anions, such as catalyst metal salt added when making

0025]As for an organic polymer (A), what replaced a hydrogen atom in a hydroxyl group of a hydroxyl 0026]As for the number of hydroxyl groups per molecule of a polyoxyalkylone polymer (F) used for group content polyoxyalkylene polymer (F) by a formula (1) is preferred. carbonate combination etc. form, and cations are included.

physical properties, such as viscosity, intensity, and clongation, that they are especially 2-4 pieces 2his invention, it is preferred that thay are 2-10 pieces. It is especially preferred from balance of

0027]Desirable polymera (F) are polyoxypropylene dlol, polyoxypropylena triol, and polyoxypropylene etraol. When using for a method of the following (b) or (**), unsaturation group end polyoxyalkylena 0028]As for a hydroxyl value conversion molecular weight of a polyoxyalkylone polymer (F), 5000noncoars, such as polyoxypropylene giycol monoallyl ether, can also be used.

weight calculated by a product of a functional group number of an initiator, and a molecular weight 0029]A hydroxyl value conversion molecular weight of this invention says a thing of a molecular ser hydroxyl group of polyoxyalkylene of a polymer used when manufacturing a polyoxyalkylene 30000 are preferred, and 8000-30000 are more preferred.

solymer (F) containing terminal hydroxyl groups.

0030]An organic polymer (A) has a hydrolytic silicon group expressed with a formula (1).

'0032]As for R', a hydroxyl group or a hydrolytic basis, and a of substitution of the carbon numbers -20 or an unsubstituted univalent hydrocarbon group, and ${\rm R}^2$ are the integers of 1-3 a divalent 0031]-R2-SIX,R1 3-s - (1)

organic group and X among a formula.

group, a phenyl group, and a fluoro alkyl group preferably. They are a methyl group, an ethyl group, a [0033]R in a formula (1) is a divalent organic group. R¹ is a univalant hydrocarbon group substitution of the carbon numbers 1-20, or unsubatituted, and are a with a oarbon number of aight or leas alkyl propyi group, a butyl group, a hexyl group, a cyclohexyl group, a phenyl group, etc. especially

which has a carbon atom among these, four or less are [six or less] preferred. As desirable X, a with 0035]Next, a manufacturing method of an organic polymer (A) is explained. The organic polymer (A) [0034]X is a hydroxyl group or a hydrolytic basis, and is a halogen atom, an alkoxy group, an acyloxy group, an amide group, an amino group, an aminooxy group, a KETOKISHI mate group, and a hydride compound is liquefied at a room temperature, and when a cured body holds pliability and uses for a a carbon number of four or less lower alkoxy group especially a methoxy group, an ethoxy basis, a this invention can introduce a hydrolytic silicon group into an end of a hydroxyl group content group as a hydrolytic basis, for example. As for especially a carbon number of a hydrolytic basis scaling material, adhesives, etc. also at low temperature comparatively, it is provided with the olyoxyalkylene polymer (F) by a method which is stated to the following (**) - (**). Such a propoxy group, etc. can be illustrated, a is an integer of 1-3 and 2 or 3 is preferred.

0038](**) A method to which a silicon hydride compound expressed with a terminal unsaturated roup introduction thing (G) and a formula (3) of a polymer (F) is made to react.

JP.09-012860,A [DETAILED DESCRIPTION]

0037]HSiX_aR¹ 3-2 -- (3)

After setting terminal hydroxyl groups OH of a polymer (F) to OM (M ia an alkaline metal), There is a oond, a urethane bond, carbonate combination, etc. When polymerizing alkylene oxide in manufacture parrying out copolymerization of the unsaturation group content alkylene oxide, such as allyl glycidyl 0039]As a method of obtaining a terminal unsaturated group introduction thing (G) of a polymer (F), nalogenated hydrocarbon, such as an allyl chloride, react to a polymer (F), and combining by ester of a polymer (F). It is obtained also by using a terminal unsaturated group content mono-hydroxy method of making a compound which has a functional group which can react to a method or an unsaturation group, and a hydroxyl group which ara made to react to unsaturation group content compound as a method of introducing an unsaturation group into a side chain, or an initiator by 0038]However, Riin formula, X, and a ara tha same as tha above.

[0041](**) A method to which W basis of a silicon compound expressed with a formula (4) to this [0040](**) How to make a polymer (F) react to a compound which has a hydrolytic silicon group isocyanate group is made to react after making polyisocyanate compounds, such as tolylene diisocyanate, react to a polymer (F) and considering it as an isocyanate group end. expressed with an isocyanate group and a formula (1).

thing (G) of a polymer (F) and a sulfhydryl group of a silicon compound expressed with a formula (4) and the active hydrogen containing group as which W was chosan from a hydroxyl group, a carboxyl 0043]however, R^{tin formula} . X, and a -- the above -- the same -- R⁵ are a divalent organic group 0044j(**) A method to which an unsaturation group of a terminal unsaturated group introduction group, a suifhydryl group, and an amino group (the 1st class or the 2nd class). 0042]R¹ ₃₋₄-SiX₄-R⁵W ... (4)

0045JAs for removal of an ionic impurity. It is preferred to carry out in suitable stages before making whose W is a sulfhydryl group are made to react.

0046]After making into an insoluble salt intrinsically an ionic impurity contained in a polymer (F) to a polymer (F), After an ionic impurity contained in a polymer (F) by removing this salt from a polymer each silicon compound react in each procedure of the above-mentioned (b) - (**), and it sets the (F) shall be 50 ppm or less, a hydrolytic sificon group is introduced into a polymer (F), and it is otal amount to 50 ppm or less. That is, a following method can be illustrated. considered as an organic polymer (A).

0048]A molecular weight of an organic polymar (A) in this invention is computed based on a hydroxyl (0047] An ionic impurity contained in a terminal unsaturated group introduction thing (G) of a polymer terminal unsaturated group introduction thing (G) as an insoluble salt intrinsically shall be 50 ppm or less, it is made to raact to a allicon hydride compound expressed with a tarminal unsaturated group introduction thing (Q) and a formula (2), and is considered as an organic polymer (A). removing this salt from a terminal unsaturated group introduction thing (G) after considering it in a molecular weight, 5000-30000 are preferred. If elongation will become hard low in a cured body if (F), After an ionio impurity contained in a terminal unsaturated group introduction thing (G) by valua conversion value molecular weight of a polymer (F) which is a raw material. As for this

ower than 5000, and 30000 is exceeded, pliability and clongation of a cured body are satisfactory, but

viscosity of the polymer itself [this] becomes remarkably high, and practicality becomes low.

Especially 8000-30000 are preferred.

0049][Tin catalyst] A ouring catalyst (B) and an organic amine compound (C) which are chosen from invention are used. Although these metal carboxylate itself acts to promote hardening of an organic polymer (A), it is insufficient for the purpose of this invention, and it is indispensable to use together carboxylate of divalent tin, carboxylate of divalent bismuth, and carboxylate of divalent lead in this with an organic amine compound (C).

iaurio acid, stearic acid, etc. A curing catalyst (B) may be a mixture. [0051]Especially the amount of these metal carboxylate used has 0.01 – 3 preferred weight section numbar of 20 or less alighatic carboxylic acid, there are heptancic soid, octylic acid, decanoic acid, 0050]As for a curing catalyst (B), it is preferred that it is a salt of with a carbon number of 20 or less of straight chain shape or a letter of branching aliphatic carboxylic soid. As with a carbon 3.01 to 10 weight saction to organic polymer (A) 100 weight saction. nttp://www4.ipdl.inpit.go.jp/cgi-bin/tran_web_cgi_ejje?atw_u=http%3A%2F%2Fwww4.ipdl.i.. 2010/04/30

nttp://www4.ipdlinpit.go.jp/ogi~bin/tran_web_ogi_ejje?atw_u=http%3A%2F%2Fwww4.ipdl.L. 2010/04/30

nonoamine or with a carbon number of 20 or less polyamine is preferred. A compound which has a 0052]As an organic amine compound (C) of this invention, with a carbon number of 20 or less hydrolytic silicon group expressed with an amino group and a formula (2) to intramolacular is

.0053]-R3-SIX1 BR4 3-5 -- (2)

ubstitution of the carbon numbers 1-20 or an unsubstituted univalent hydrocarbon group, and X¹ are 0054]As for a divalent organic group and R^4 , a hydroxyl group or a hydrolytic basia, and b of R³] the integers of 1-3 among a formula.

number of 20 or less aliphatic series mono(or poly) amine, with a carbon number of 20 or less alioycle 0055]As with a carbon number of 20 or less amine, it is preferred to be chosen out of with a carbon fellows mono(or poly) amine, and with a carbon number of 20 or less aromatic mono(or poly) amine for example. Specifically, the following can be illustrated.

0056]Monoamine: Methylamine, ethylamine, propylamine, isopropylamine, A butylamine, amyl amine, exylamine, octylamine, nonyl amine, Lauryl amine, dimethylamine, diethylamine, dipropyl amine, Disopropylamine, dibutyl amine, diamylamine, trimethylamine, Triethylamine, tripropylamine.

tributylamine, triamylamine, Cyclo propylamine, a cyclo butylamine, cyclopentyl amine, cyclopentyl amine, oycloboxylamine, nN-diethylamiline, NN-dimethylamiline, nN-diethylamiline, a oluidine, benzylamine, diphenylamine, etc.

0057]Polyamine: Ethylonediamine, diethylenediamine, triethylenediamine, Hexamathylenediamine, sodeoamethylenediamine, diethylenetriamine, Triethylenetatramine, tetraethylenepentamine, lethanolamine, triethanolamine, N.N.N.N.-tetramethyl 1,3-butanodiamine, N.N.N.N.-

0058]As a compound which has a hydrolytic silicon group expressed with an amino group and a ormula (2) to intramolecular, Especially a reactant with an organic amine compound which has a ydrolytic silicon group in intramolecular known as amino group content Silang, and a compound etramethylethylenediamine, etc.

0059]As amino group content Silang, specifically gamma-aminopropyl trimethoxysilane, N-(betawhich has an epoxy group and a hydrolytic silicon group in intramolecular known as amino group aminoethyl)-gamma-aminopropyl trimethoxysilane, gamma-aminopropyl triethoxysilane, N-(beta-Ilmethoxysilane, N-(beta-aminoethyl)-gamma-aminopropyl methyl dimethoxysilane, gammaaminoethyl)-gamma-aminopropyl triethoxysilane, There are gamma-aminopropyl methyl content Silang and epoxy group content Silang is preferred.

nvention -- 0.01 - **** for 10 weight sections - things are preferred. 0. Especially one to 3 weight .0080]As epoxy group content Silang made to react to these amino group content Silang, gamma-0061]receiving organic polymer (A) 100 weight section in an organic amine compound (C) in this zlycidyloxypropyi trimethoxysllane, There are gamma-glycidyloxy propyl triethoxysilane, gammaglycidyloxy propylmethyl dimethoxysilane, gamma-glycidyloxy propylmethyl diethoxysilane, etc. nethyldiethoxysilane. section is preferred.

uminopropyl methyldiethoxysilane, and N-(beta-aminoethyl)-gamma-aminopropyl

used, 50 to 250 % of the weight is preferred zero to 1000% of the weight to an organic polymer (A). The following are mentioned as an example of a bulking agent. These bulking agents may be used (0062]A bulking agent (D) is used in this invention. As for especially the amount of bulking agent

independently and may be used together two or more sorts. [D063]An example of a bulking sepert : Galcium carbonate, fumes silica, a edimentation nature silica, a silicio add antyvide, lydrous silicio addidantyvide, lydrous silicio addidantico. active white, a milt balloon, wood flour, pulp, a cotton chip, mice, and blacking wash farina --- rubbing Calcination clay, clay, talo, titanium oxide, bentonite, organio bentonite. Ferrio oxide, a zino oxide, an fibrous fillers, such as powder state bulking agents, such as farina, graphite, aluminum impalpable

cowder, and the Flint powder, asbestos, glass fiber, a glass filament, carbon fiber, the Kevlar textiles, [0064]In this invention, a plasticizer can be used arbitrarily. As a plasticizer, dioctyl phthalate, dibutyl Dioctyl adipate, Alcohol ester, such as aliphatic-carboxylic-soid ester; pentaerythritol ester, such as succinic soid diisodecyl, dibutyl sebacate, and butyl oleate; Trioctyl phosphate, Phosphoric ester, ohtalate, phthalio aoid diisononyl ester. Phthalic ester, such as phthalic acid benzyl butyl ester; and a polyethylene fiber.

http://www4.jpdl.inpit.go.jp/cgi-bin/tran_web_ogi_ejje?atw_u=http%3A%2F%2Fwww4.jpdl... 2010/04/30

4,5-apoxy hexahydrophthelic soid dioctyl, and epoxy stearic soid benzyl, can be independent, or can such as tricresyl phosphate; epoxy plasticizer; chlorinated paraffins, such as epoxidized soybean oll, use it with two or more sorts of mixtures.

0065]The constituent of this invention can contain still more publicly known various additive agents resin and an epoxy resin, and hydrogenation castor oil, paints, various kinds of antiaging agents, an etc. As an additive agant, thixotropy grant agents, such as adhesive grant agents, such as phenol ultraviolat ray absorbent, etc. can be used.

(Examples 7-14, 21-28, 35-45) explain this invention concretely below, this invention is not limited to The synthetic examples 1-7 first show the example of manufacture of the organic polymers P1-P7 which are the raw materials of an organic polymer (A) (however, P7 organic polymer for Example]Although working example (Examples 1-6, 15-20, 29-34) and a comparative example

Polyoxypropylene triol of hydroxyl value 11.2 mgKOH/g and viscosity 7000cP in 25 ** was obtained. polyoxypropylene triol, methanol was distilled off, and the organic polymer (P1) which added the allyl 0067][Synthetic example 1] Propylene oxide is polymerized with a zinc hexa cyanocobaltate glyme complex by using the glycerin propylene oxide addition of the molecular weight 1000 as an initiator, chloride, changed terminal hydroxyl groups into the allyloxy group, and contained metal salt as an Then, after adding the sodium methoxide of the equivalent 1.1 times to the hydroxyl group of comparison). A part shows a weight section.

glyme complex by having used ethylene glycol as the initiator, and the viscosity in hydroxyl value 9.3 changed terminal hydroxyl groups into the allyloxy group by the method of the synthetic example 1 0068][Synthetic example 2] Propylene oxide was polymerized with the zino hexa oyanooobaltate mgKOH/g and 25 ** obtained polyoxypropylene diel of 8000cP. The organic polymer (P2) which description, and contained metal salt as an impurity was obtained. impurity was obtained.

thme complex by having used ethylene glycol as the initiator, and the viscosity in hydroxyl value 5.6 0070][Synthetic example 4] Propylene oxide is polymerized with a zinc hexa cyanocobaltate glyme complex by using the pentaerythritol propylene oxide addition of the molecular weight 1000 as an 0089][Synthetic example 3] Propylene oxide was polymerized with the zinc hexa cyanocobaltate mgKOH/g and 25 ** obtained polyoxypropylene diol of 17000cP. Then, the organic polymer (P3) which changed terminal hydroxyl groups into the allyloxy group by the method of the synthetic example 1 description, and contained metal salt as an impurity was obtained.

initiator. The viscosity in hydroxyl value 13.2 mgKOH/g and 25 ** obtained polyoxypropylene tetraol of 6000oP. The organic polymer (P4) which changed terminal hydroxyl groups into the allyloxy group of 6000oP.

y the method of the synthetic example 1 description, and contained metal salt as an impurity was

8, and the polyoxypropylene triol obtained in the synthetic example 1 by the waight ratio 2 to 1, the organic polymer (P5) which changad terminal hydroxyl groups into the allyloxy group by the method of [0071][Synthetic example 5] After mixing the polyoxypropylenc diol obtained in the synthetic example '0072][Synthetio example 6] After mixing the polyoxypropylene diol obtained in the synthetic example method of the synthetic example 1 description, and contained metal salt as an impurity was obtained and the polyoxypropylene tetraol obtained in the synthetic example 4 by the weight ratio 4 to 1, the organic polymer (P8) which changed terminal hydroxyl groups into the allyloxy group by the the synthetic example 1 description, and contained metal salt as an impurity was obtained.

making 3-mol chlorobromomethane react under alkali existence, the organic polymer (97) which added the allyl chloride, changed terminal hydroxyl groups into the allyloxy group, and contained motal polyoxypropylene diol of the hydroxyl value conversion molecular weight 3000 is received. After 0073][Synthetic example 7] In accordance with a method given in JP,59-25808,B, 4 mol of saft was obtained.

[0075]Then, by the publicly known method using a platinum catalyst, tha mathyl dimethoxy silyl propyl 0074][Examples 1-14] The organic polymers P1-P7 were refined by purification method A - 1, and the amount of residual metal ions (unit: ppm) was measured. The viscosity (unit: oP) of the organio group was introduced into the organic polymer tarminal, and the organic polymers \$1-\$14 were oolymers P1-P7 after refining is also collectively shown in Tables 1-2.

http://www4.ipdl.inpit.go.jp/ogi-bin/tran_web_ogi_ejje?atw_u=http%3A%2F%2Fwww4.ipdli... 2010/04/30

7/9 ページ JP,09-012860,A [DETAILED DESCRIPTION]

between the therman detective viscolety of the operation polymers of 1:51 fit is also shown in a table, consists of the methy dimethory slid propyl group as an end group is the rarge of organic concents of the methy dimethory slid propyl group as an end group is the rarge of organic colopymer I gen to CI Immod. I Thi manufact forsteaded match loss of the debtafield organic colopymers 11:54 was not different from hebrico methyd imethory slid propyl group introduction.

If Mydrogenstein castor old, once oncy of phenotic antioxidant, 0.5 coay of ultraviolet ray absorbent, and the Coay of ultraviolet ray absorbent, and the Coay of ultraviolet properties of the Coay of ultraviolety and ultr

0076][Examples 15-28] 160 copies of calcium carbonate, 60 copies of dioctyl phthalate, five copies

Job 1 John was contentanta, to sen copies or metal cancopyage snown in the Table or night column, three topies or fall of 20 copies of discyl phthalate, and 60 copies of calcium carbonate were meeded, and it was considered as the hardening agent (K1 – K5).

00793The viscousty (after-atenge viscosity) (unit of 9 after atenge to 14 days at the viscosity 00793The viscosity (after-atenge viscosity) (unit of 9) after atenge viscosity (unit of 90 after atenge viscosity) (unit of 90 after annufacture of base resin and 50 as was measured. A resilt is aftern in 70 after 90 after

1000 [Enemption 23-44] (to copies of base with (LH-M4) and 8.8 copies of partnering agents (K1 – 1000) with a mean shown in the table solew were honeign that it might become a thickness of 3 on into the one of a cylindrical shape 4 on in diameter, and it was neglected under the sunspine of 10° and 65% buming to 60° burn. It has itsulation of more area may be an experimental to each of the standard of the sunspine of 10° and 65% burns, the standard of a standard when the standard of the standard of the sunspine of 10° and 65% burns, the standard of the standard beautiful in false 5.7. The one where paretrains is larger means that hardening from the sunspine of the paretrains is larger means that hardening

ollycollycinistation modes A) The aboyocomplete potentiar Egit towards Egit of settle discussional dilipidosean pyrabologistation of the molecular weight (1000 to which 10 to 6 the weight of endual dilipidosean pyrabologistation of the abolite of the discussion of the old of the control metal settle decis, as an inputry, and it adjusted as (90 ± for 1 lour. Thus, after populars which contain metal settle decis as in inputry, and it adjusted as (90 ± for 1 lour. Thus, after properties of the control metal settle decis are inspurity, and it adjusted as (90 ± for 1 lour.) Thus, after discussion of the control metal settle decis and the competence of the control of the control

Visit and medical things were obtained by the question between the sets as an impurity of the sets and the sets as an impurity of the sets of the sets

decompression, and refining things were obtained [0082] Trable 11

[Table 1]

14	-	9	•	=		•	
原料整合体	P.1	P 2	E d	P 4	P 5	P 6	
報覧供	۲	۲	٨	٨	۲	٨	
精製後指度	6500	7500	15000	5500	12000	13000	
現存イオン量							
Na m	60	4	63	2	69	e	
Zo. 42	187	157	1以下	1以下	187	187	
₩º3	LAT	187	187	187	1 R.F	1 B/F	
CI- ##	ıo	ນາ	8	80	un	ıs	
有機關合体	81	62	83	S 4	25	86	
数指统指数	8000	800	17000	7500	13000	13500	

[0083]

http://www4.ipdl.inpit.go.jp/ogi-bin/tran_web_ogi_ejje?atw_u=http%3A%2F%2Fwww4.ipdl.i.. 2010/04/30

[Table 2]

	ł
	ŀ
_	Ì
Table 2	-
Table 2	

製料製金 P1	٦,		•	=	7.1	•	7
整数形	-	P 2	80	P.4	P 5		P 7
		~	۲	+	4	+	~
特型设施度(6500	7500	15000	2500	12000	13000	9008
製作イオン最							
	2 5	5 2	3 2	3 3	26	38	2
25.25	2	60	60	ED	4	ц	0
₩. ₁ 03	~	4	00	6	4	ß	0
	3 5		40	4 5	35	44	4
右接重合体 S	88		810	S 1 1	\$12	\$13	\$14
製造後指成	9000	10000	19000	8500	13000	16000	

[Table 3]

£	1.2	16	1 2	80	61	2 0	2 1	
か神器会体	18	6 6	60	4.8	200	9.5	8	
被	M	22	M3	M4	M6	W 6	M7	
	1.575	167		137	173	20万	177	
野戲後指皮	167	163		133	187	217	35万	

[0085] [Table 4]

E	2.2	23	2.4	2 5	26	2.2	80
有機重合体	8.8	\$ 1.0	\$11	812	S 1 3	8.7	814
裹州	876	M9	M10	X11	M12	M13	M14
製造後粘度	1875	9	167	19万	20万	173	17万
貯敷後粘度	457	553	87.H	407	523	187	18万

[0086] [Table 5]

\$		29	30		60	33	3.4
#	篠	N I	M2	K3	M4	S N	Me
製化を	· 165	KI	K1	X.	K 2	К3	M M
类	*	0.7	0.7	0.5	0.8	0. 7	0. 5

[0087] [Table 6] http://www4.ipdl.inpit.go.jp/ogi-bin/tran_web_ogi_ejje?atw_u=http#3A%2F%2Fwww4.ipdl.i. 2010/04/30

6	M12 K4 0.4
3.9	M11 K3 0.5
3.8	M10 K2 0.7
3.7	M9 K1 0.4
3.6	M8 K1 0.6
3.5	M7 K1 0.6
#	世 類化剤 鮮入度

[0088] [Table 7]

43 42 41

4 5 M3 K6 2. 1

[0089] [Table 8]

政化組合布する金属カルボン酸塩 オクテル酸スズ (2価)

オクチル酸ピスマス (2個) (長微化成社コスキャット83) パーサチック酸ピスマス (2個) (The Shepherd Chemical 社類BiSMUTE VERSALATE) ネオデカン酸ビスマス (2価) (日本化学産業製ニッカオクチックススズ) K4 X X

(The Shepherd Chemical AMMERISAUTH NEGRECANOATE) ジブチルスズジラウレート (4価) K S [0390] [Effect of the Invention]Storage stability is improved remarkably and the hardenability constituent of this invention has the good hardenability of the depths.

[Translation done.]

* NOTICES *

langes caused by the use of this translation. PO and INPIT are not responsible for any

This document has bean translated by computer. So the translation may not reflect the original

**** shows the word which can not be translated. 3. In the drawings, any words are not translated.

CORRECTION OR AMENDMENT

Kind of official gazette] Printing of amendment by regulation of Patent Law Article 17 of 2 Publication date Heisei 11(1999) (1999) November 26 Section Type] The 3rd Type of the part III gate

Annual volume number] Publication of patent applications 9-129 Date of Publication]Haisai 9(1997) (1997) January 14 Publication No.JJP,9-12860.A

Application number]Japanese Patent Application No. 7-164188 International Patent Classification (6th Edition)]

308L 71/02 LDC

308K 5/09 KAR

5/17 KAY

COBL 101/10 [F1]

D08L 71/02 LDC COBK 5/09 KAR 717 KAY

COST 101/10

Filing date] Heisei 11(1999) March 10 Written Amendment Amendment 1

Document to be Amended Description |tem(s) to be Amended]Claims Method of Amendment]Change

Claim 1]A curing catalyst (B) chosen from the following organic polymer (A), carboxylate of divalent Proposed Amendment Claim(s)

in, carboxylate of divalent bismuth, and carboxylate of divalent lead, and an organic amine compound by polymerizing alkylene oxide in an initiator by making a composite metal cyanide complox (E) into a catalyst, An organic polymer (A) whose total amount of an ionic impurity it has a hydrolytic silicon Organic polymer) It is derived from a hydroxyl group content polyoxyalkylene polymer (F) produced (C) and a room-temperature-curing nature constituent containing a bulking agent (D).

group expressed with a formula (1), and is 50 ppm or less. -R2-SIX,R1 3-3 ... (1)

an unsubstituted univalent hydrocarbon group, and R² are the integers of 1–3 a divalent organic group As for R1, a hydroxyl group or a hydrolytic basis, and a of substitution of the carbon numbers 1-20 or

nttp://www4.ipdkinpit.go.jp/ogi-bin/tran_web_ogi_ejje?atw_u=http%3A%2F%2Fwww4.ipdl.i.. 2010/04/30

and X among a formula.

[Qiaim 2]The room-tamperatura-curing nature constituent according to claim 1 whose organic amine compound (C) is a compound which has with a carbon number of 20 or leas amine or a hydrolytic silicon group exprassed with an amino group and a formula (2) to intramolecular. bR* 3-b -- (2) -R3-SIX1

パ2ページ

As for a divalent organic group and R^4 , a hydroxyl group or a hydrolytic basis, and $\mathfrak b$ of substitution of Claim 3]The room-temperature-curing nature constituent according to claim 1 or 2 which is an ionic the carbon numbers 1-20 or an unsubstituted univalent hydrocarbon group, and X1 are [R3] the integers of 1-3 among a formula.

Claim 4]An organic polymer (A) an ionic impurity contained in a polymer (F), After an ionic impurity impurity in which an ionic impurity contains metallic compounds and/or an alkali metal compound resulting from a composite metal cyanide complex (E).

contained in a polymer (F) by removing this salt from a polymer (F) after considering it to a polymer (F) as an insoluble ash intrinsically shall be 50 ppm or less. The room-tempereture-ouring nature constituent according to any one of cleans it of a which is an organic polymer produced by introducing intyochio silicon group into a polymer (F). Claim 5]An organic polymer (A) an ionic impurity contained in a terminal unsaturated group

selt intrinsically shall be 50 ppm or less, The room-temperatura-curing nature constituent according sydride compound expressed with a terminal unsaturated group introduction thing (G) and a formula ntroduction thing (G) of a polymer (F). After an ionic impurity contained in a terminal unsaturated thing (G) after considering it in a tarminal unsaturated group introduction thing (G) as an insoluble group introduction thing (G) by removing this salt from a terminal unaaturated group introduction to any one of claims 1 to 3 which is an organic polymer obtained by making it react to a silicon

substitution of the carbon numbers 1-20 or an unsubstituted univalent hydrocarbon group, and X. [Claim 6]The room-temperature-ouring nature constituent according to any one of claims 1 to 5 A hydroxyl group or a hydrolytic basis, and a of R¹ are the integers of 1-3 among a formula whose hydroxyl value conversion molecular weights of a polymer (F) are 5000-30000.

[Translation done.]